

(10) **Patent No.:** US 9,455,138 B1
(45) **Date of Patent:** Sep. 27, 2016

- (54) **METHOD FOR FORMING DIELECTRIC FILM IN TRENCHES BY PEALD USING H-CONTAINING GAS**
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- (*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

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- (21) Appl. No.: **14/937,053**
(22) Filed: **Nov. 10, 2015**

USPTO; Office Action dated Aug. 27, 2010 in U.S. Appl. No. 12/118,596.

(Continued)

- (51) **Int. Cl.**
H01L 21/311 (2006.01)
H01L 21/02 (2006.01)
- (52) **U.S. Cl.**
CPC *H01L 21/0228* (2013.01); *H01L 21/02123*
(2013.01); *H01L 21/02208* (2013.01)
- (58) **Field of Classification Search**
CPC H01L 21/0228; H01L 21/02123;
H01L 21/02208; H01L 21/0262; H01L
21/02642; H01L 21/02219; H01L 21/3141;
C23C 16/277; C23C 16/45525
USPC 438/680, 700, 142, 474, 475, 513, 515,
438/743, 744, 769, 791; 257/E21.006,
257/E21.054, E21.17, E21.267, E21.278,
257/E21.293, E21.311, E21.319, E21.321,
257/E21.327
See application file for complete search history.

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(57) **ABSTRACT**

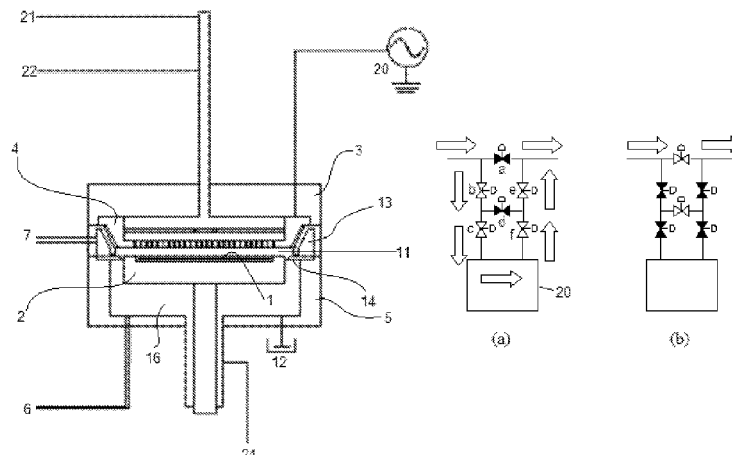
A method for forming a dielectric film in a trench on a substrate by plasma-enhanced atomic layer deposition (PEALD) performs one or more process cycles, each process cycle including: (i) feeding a silicon-containing precursor in a pulse; (ii) supplying a hydrogen-containing reactant gas at a flow rate of more than about 30 sccm but less than about 800 sccm in the absence of nitrogen-containing gas; (iii) supplying a noble gas to the reaction space; and (iv) applying RF power in the presence of the reactant gas and the noble gas and in the absence of any precursor in the reaction space, to form a monolayer constituting a dielectric film on a substrate at a growth rate of less than one atomic layer thickness per cycle.

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20 Claims, 2 Drawing Sheets



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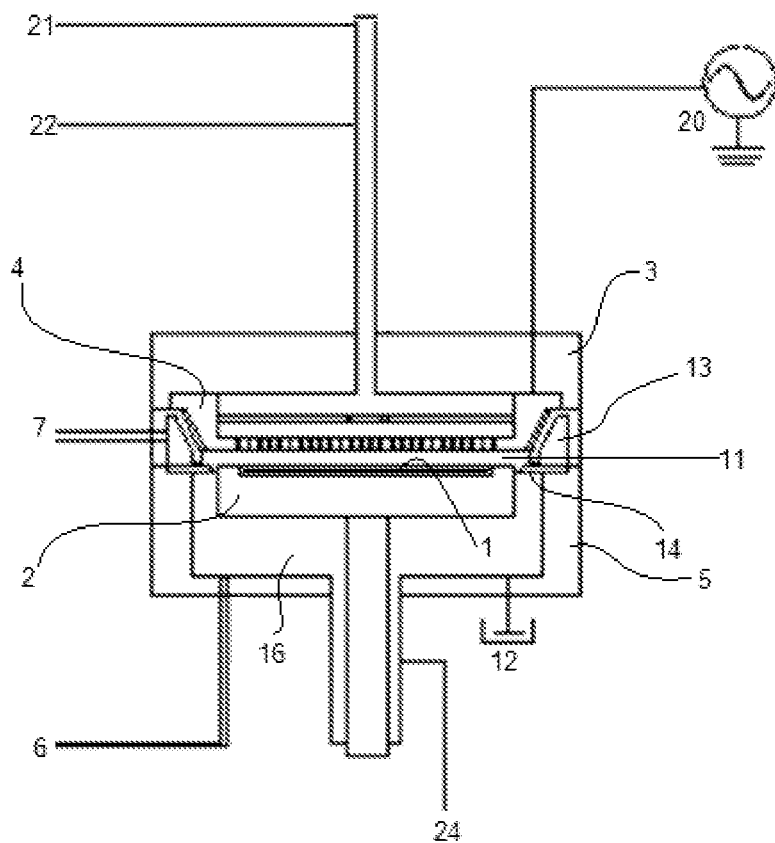


Fig. 1A

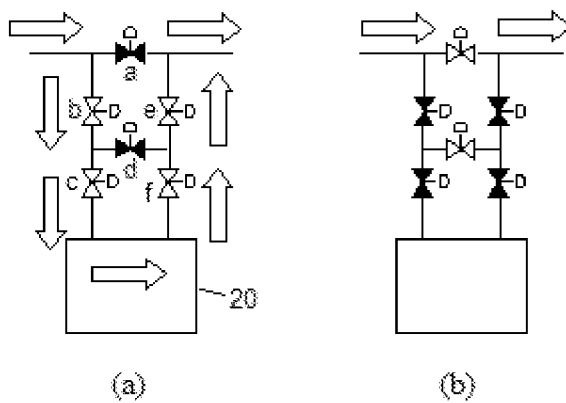


Fig. 1B

Fig. 2

Parameter	Feed	Purge	RF	Purge
Precursor				
H ₂				
Carrier				
Dilution				
RF				

Fig. 3

Parameter	Feed	Purge	RF-1	Purge	RF-2	Purge
Precursor						
H ₂						
Carrier						
Dilution						
RF						

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METHOD FOR FORMING DIELECTRIC FILM IN TRENCHES BY PEALD USING H-CONTAINING GAS

BACKGROUND

1. Field of the Invention

The present invention generally relates to a method for depositing a dielectric film in a trench of a substrate by plasma-enhanced atomic layer deposition (PEALD). The present invention also relates to a method for increasing a sidewall coverage of a dielectric film deposited by PEALD.

2. Related Art

As a method of depositing a film having a good step coverage, atomic layer deposition (ALD) using chemisorption of a precursor is commonly performed. In this method, a film deposits more evenly in trenches of semiconductor circuits than does a film by CVD or the like. However, in plasma-enhanced atomic layer deposition (PEALD), since a sidewall of a trench of a substrate undergoes less ion bombardment than does a top surface of the substrate, surface reaction at the sidewall is less active than on the top surface, causing a problem that an etch rate of a film at the sidewall is different from (higher than) that on the top surface. In particular, when a precursor has an adsorption inhibition problem due to e.g., the presence of hydrocarbon components in the molecule of the precursor, the step coverage of a film deposited on a sidewall becomes low (e.g. 40% or less).

Conventionally, by increasing the process temperature or the like, the quality of a dielectric film (e.g., density, hardness) deposited on a sidewall is improved so that the etch rate at the sidewall can be decreased. However, the improvement is partial, and the problem in different etch rates between the sidewall and the top surface is not sufficiently resolved.

Any discussion of problems and solutions in relation to the related art has been included in this disclosure solely for the purposes of providing a context for the present invention, and should not be taken as an admission that any or all of the discussion was known at the time the invention was made.

SUMMARY

In some embodiments of the present invention, the thickness of film deposited on a target side of a trench of semiconductor circuits (substrate) can be controlled. In some embodiments, the thickness of film deposited on a sidewall of a trench relatively increases, i.e., the thickness of film deposited on a top (blanket) surface of the substrate relatively decreases. In one approach, the thickness of film on a sidewall of a trench can be controlled by controlling chemisorption of a precursor on a surface using a chemisorption-inhibitor gas to which the substrate is exposed as a preliminary treatment before depositing a film thereon, wherein functional groups exposed on the top surface of the substrate are terminated by Si—H bonds using a chemisorption-inhibitor gas such as hydrogen gas, thereby interfering with chemisorption of the precursor on the top surface and relatively increasing the deposition rate of film on the sidewall. In some embodiments of the present invention, another approach other than the above approach is taken, wherein when depositing a silicon-based dielectric film such as a SiC or SiN film by PEALD, a hydrogen-containing gas is used as a reactant gas so as to cause not only deposition of a film but also etching of the film by a plasma. Since more species excited by a plasma reach a flat

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surface, i.e., a top surface of a substrate and a bottom of a trench of the substrate, than excited species reaching a sidewall of the substrate, by controlling process parameters including feed quantity of a precursor and intensity and duration of a hydrogen plasma, controlling and balancing deposition and etching of film can be accomplished predominantly on a flat surface, whereby the thickness of film at the sidewall of the trench can be adjusted relative to the thickness of film on the flat surface of the substrate (the sidewall receives less effect of a plasma than does the flat surface). In the above, a film is being deposited by excited species of precursor while being etched by hydrogen and argon plasma, for example.

For purposes of summarizing aspects of the invention and the advantages achieved over the related art, certain objects and advantages of the invention are described in this disclosure. Of course, it is to be understood that not necessarily all such objects or advantages may be achieved in accordance with any particular embodiment of the invention. Thus, for example, those skilled in the art will recognize that the invention may be embodied or carried out in a manner that achieves or optimizes one advantage or group of advantages as taught herein without necessarily achieving other objects or advantages as may be taught or suggested herein. Further aspects, features and advantages of this invention will become apparent from the detailed description which follows.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features of this invention will now be described with reference to the drawings of preferred embodiments which are intended to illustrate and not to limit the invention. The drawings are greatly simplified for illustrative purposes and are not necessarily to scale.

FIG. 1A is a schematic representation of a PEALD (plasma-enhanced atomic layer deposition) apparatus for depositing a dielectric film usable in an embodiment of the present invention.

FIG. 1B illustrates a schematic representation of a precursor supply system using a flow-pass system (FPS) usable in an embodiment of the present invention.

FIG. 2 illustrates a PEALD process sequence according to an embodiment of the present invention.

FIG. 3 illustrates a PEALD process sequence according to another embodiment of the present invention.

DETAILED DESCRIPTION OF EMBODIMENTS

In this disclosure, “gas” may include vaporized solid and/or liquid and may be constituted by a single gas or a mixture of gases. Likewise, an article “a” or “an” refers to a species or a genus including multiple species. In this disclosure, a process gas introduced to a reaction chamber through a showerhead may be comprised of, consist essentially of, or consist of a precursor and a reactant gas. The reactant gas may include a gas involving oxidizing and/or nitriding the precursor when RF power is applied to the reactant gas. The reactant gas can be introduced continuously to a reaction space if it is not reactive to the precursor without RF power. The precursor can be introduced with a carrier gas such as a noble gas. A gas other than the process gas, i.e., a gas introduced without passing through the showerhead, may be used for, e.g., sealing the reaction space, which includes a seal gas such as a noble gas. In some embodiments, “film” refers to a layer continuously extending in a direction perpendicular to a thickness direction

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substantially without pinholes to cover an entire target or concerned surface, or simply a layer covering a target or concerned surface. In some embodiments, "layer" refers to a structure having a certain thickness formed on a surface or a synonym of film or a non-film structure. A film or layer may be constituted by a discrete single film or layer having certain characteristics or multiple films or layers, and a boundary between adjacent films or layers may or may not be clear and may be established based on physical, chemical, and/or any other characteristics, formation processes or sequence, and/or functions or purposes of the adjacent films or layers. Further, in this disclosure, any two numbers of a variable can constitute a workable range of the variable as the workable range can be determined based on routine work, and any ranges indicated may include or exclude the endpoints. Additionally, any values of variables indicated (regardless of whether they are indicated with "about" or not) may refer to precise values or approximate values and include equivalents, and may refer to average, median, representative, majority, etc. in some embodiments. The terms "constituted by" and "having" refer independently to "typically or broadly comprising", "comprising", "consisting essentially of", or "consisting of" in some embodiments.

In the present disclosure where conditions and/or structures are not specified, the skilled artisan in the art can readily provide such conditions and/or structures, in view of the present disclosure, as a matter of routine experimentation.

In all of the disclosed embodiments, any element used in an embodiment can be replaced with any elements equivalent thereto, including those explicitly, necessarily, or inherently disclosed herein, for the intended purposes. Further, the present invention can equally be applied to apparatuses and methods.

In this disclosure, any defined meanings do not necessarily exclude ordinary and customary meanings in some embodiments.

In some embodiments, the term "precursor" refers generally to a compound that participates in the chemical reaction that produces another compound, and particularly to a compound that constitutes a film matrix or a main skeleton of a film, whereas the term "reactant" refers to a compound that activates a precursor, modifies a precursor, or catalyzes a reaction of a precursor.

The dielectric film includes, but is not limited to, a low-k film constituted by a silicon carbide such as SiC, SiCN, and SiCON, a silicon oxide such as SiO, or a silicon nitride such as SiN, having a dielectric constant of about 3 to 6, typically about 3.5 to 4.5. In some embodiments, the dielectric film is formed in trenches, vias, or other recesses including side walls and bottom surfaces (collectively referred to as "trenches") by plasma-enhanced ALD or other plasma-assisted cyclic deposition methods. The trench may have a depth of about 10 nm to about 1,000 nm, typically about 100 nm to about 500 nm, and an aspect ratio of about 1 to about 10, typically about 2 to about 5 (e.g., a trench having a width of about 30 nm, a depth of about 110 nm, and an aspect ratio of about 4, formed as a pattern in a silicon substrate). The thickness of the deposited dielectric film may be in a range of about 2 nm to about 500 nm, typically about 10 nm to about 100 nm, more typically about 15 nm to about 30 nm (a desired film thickness can be selected as deemed appropriate according to the application and purpose of film, etc.).

The embodiments will be explained with respect to preferred embodiments. However, the present invention is not limited to the preferred embodiments.

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In an embodiment, in a method for forming a dielectric film in a trench on a substrate by plasma-enhanced atomic layer deposition (PEALD) performing one or more process cycles, each process cycle comprises: (i) feeding a silicon-containing precursor in a pulse to a reaction space where the substrate is placed, said precursor being constituted by one or more hydrocarbon-containing compounds selected from the group consisting of: SiH_2R_2 , $\text{Si}_2\text{H}_2\text{R}_4$, SiR_2X_2 , Si_2R_6 , SiH_3R , $\text{Si}_2\text{H}_4\text{R}_2$, SiH_2RX , $\text{C}_3\text{H}_6\text{SiH}_2$, $\text{C}_2\text{H}_4\text{SiH}_2$, $\text{C}_2\text{H}_4\text{Si}_2\text{H}_2$, $\text{SiNHHSiR}_4\text{H}_2$, SiNHHSiR_6 , and SiHX_2R , wherein each X is independently chain or cyclic C_xH_y , and each R is independently chain or cyclic C_xH_y , cyclic $\text{N}_x\text{C}_y\text{H}_z$, $\text{N}(\text{C}_x\text{H}_y)_2$, $\text{N}(\text{C}_x\text{H}_y)\text{H}$, $\text{O}(\text{C}_x\text{H}_y)$, or OH, wherein x, y, and z are integers (e.g., x is an integer of 1 to 5, y is an integer of 1 to 10, and z is an integer of 3 to 15); (ii) supplying a hydrogen-containing reactant gas to the reaction space at a flow rate of more than about 30 sccm but less than about 800 sccm (e.g., about 50 sccm to about 500 sccm) in the absence of nitrogen-containing gas; (iii) supplying a noble gas to the reaction space; and (iv) applying RF power to the reaction space in the presence of the hydrogen-containing reactant gas and the noble gas and in the absence of any precursor in the reaction space, to form a monolayer constituting a dielectric film on a substrate at a growth rate of less than one atomic layer thickness per cycle. The growth rate per cycle or thickness of a monolayer refers to an average growth rate per cycle calculated based on the measured thickness of a deposited film and the number of cycles performed for the deposited film, or based on the total growth rate of the resultant deposited dielectric film. The one atomic layer thickness refers to a theoretical thickness of one atomic layer formed from a precursor gas without considering lamination or the interface between monolayers. The term "monolayer" refers to a layer formed by one process cycle of PEALD, which may not be a continuous film.

In some embodiments, the growth rate of the monolayer is less than about 0.1 nm per cycle (e.g., about 0.003 nm to about 0.09 nm). In general, the theoretical one atomic layer thickness is about 0.1 nm to about 0.5 nm, typically about 0.2 nm to about 0.3 nm. When the above-mentioned hydrocarbon-containing compound is used as a precursor, due to adsorption inhibition of the precursor by the hydrocarbon components on a substrate surface, the average growth rate of the film per cycle on a flat horizontal surface (a blanket surface) becomes less than one atomic layer thickness. In that case, in general, the growth rate of the monolayer on a sidewall is even worse than that on the blanket surface, resulting in a low step coverage on the sidewall (e.g., 40% or less). However, according to some embodiments of the present invention disclosed herein, the step coverage on the sidewall can surprisingly be increased to 80% or higher.

In some embodiments, the precursor has a chemical formula where at least one of X or R is an unsaturated hydrocarbon having, e.g., a carbon double or triple bond. In general, when the precursor includes more carbon atoms in its molecule, i.e., including fewer hydrogen atoms, more deposition of film takes place, rather than etching of film. In some embodiments, the precursor has a cyclic structure. In some embodiments, the precursor is one or more compounds selected from the group consisting of: SiH_2R_2 such as dimethylsilane, divinylsilane, and dipyrildisilane; $\text{Si}_2\text{H}_2\text{R}_4$ such as tertamethyldisilane; SiR_2X_2 such as divinyltrimethylsilane and dimethyldipyrildisilane; Si_2R_6 such as hexamethyldisilane; SiH_3R such as silylacetylene and allylsilane; $\text{Si}_2\text{H}_4\text{R}_2$ such as divinylsilane and dimethyldisilane; SiH_2RX such as vinylmethylsilane; $\text{C}_3\text{H}_6\text{SiH}_2$ such as silacyclobutane; silacycloethane; disilacycloethane;

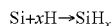
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SiNHSiR₄H₂ such as tetramethyldisilazane; SiNHSiR₆ such as hexamethyldisilazane; and SiHX₂R such as dimethyldisilane.

In some embodiments, the reactant gas is hydrogen gas. In some embodiments, the reactant gas is a hydrocarbon gas such as hexane. In some embodiments, the noble gas is argon. A combination of H₂ and Ar is most effective in counteracting deposition of film, wherein H₂ in a plasma state likely causes chemical etching whereas Ar in a plasma state likely causes physical sputtering (bombardment at an angle of about 45°) on the surface of a substrate. Thus, H₂ in a plasma state is effective to etch predominantly a film on a flat surface, whereas Ar in a plasma state is effective to etch rather uniformly a film on the flat surface and a film at the sidewall. Unlike Ar, He in a plasma state does not likely cause physical sputtering, and thus, a combination of H₂ and Ar, rather than a combination of H₂ and He, is used in some embodiments.

In some embodiments, a ratio of flow rate of the noble gas, typically Ar, to flow rate of the hydrogen-containing reactant, typically hydrogen gas, is about 5:1 to about 100:1, preferably about 10:1 to about 60:1. In the above, the flow rate of the noble gas includes a carrier gas for carrying a precursor in addition to a dilution gas. When the flow rate of the hydrogen-containing reactant, typically hydrogen gas, is too low, etching effect is not sufficient to control growth rate of film on a flat surface, whereas when the flow rate is too high, etching effect is predominant and no film is deposited on the flat surface.

When a hydrogen plasma is used, both the etching effect and the deposition effect can be obtained, and can be balanced by adjusting the flow rate of hydrogen, the number of RF pulses per cycle, the feed of a precursor, etc. The etching by a hydrogen plasma may occur as follows, for example:



On the other hand, deposition by a hydrogen plasma may occur via removing a ligand which promotes chemisorption, and via formation of dangling bonds. Further, deposition may occur through re-deposition of dissociated components by etching.

In some embodiments, in step (iii), the noble gas is supplied to the reaction space at a flow rate of about 1,000 sccm to about 5,000 sccm, preferably about 2,000 sccm to about 4,000 sccm.

In some embodiments, the reactant gas is supplied continuously to the reaction space throughout each process cycle. In some embodiments, the noble gas is continuously supplied to the reaction space throughout the process cycle.

In some embodiments, in step (iv), no gas other than the reactant gas and the noble gas is supplied to the reaction space.

In some embodiments, in step (i), the precursor is fed in an amount of about 0.00002 g/cycle to about 0.01 g/cycle and in a pulse having a duration of about 0.1 seconds to about 1.0 seconds. Since ALD is a self-limiting adsorption reaction process, the number of deposited precursor molecules is determined by the number of reactive surface sites and is independent of the precursor exposure after saturation, and a supply of the precursor is such that the reactive surface sites are saturated thereby per cycle. In other embodiments the plasma may be generated remotely and

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provided to the reaction chamber. The feed amount of precursor can be determined depending on the molecular weight of precursor.

In some embodiments, each process cycle further comprises a purging step between steps (i) and (iv), and between steps (iv) and (i) if the process cycle is repeated. In some embodiments, in step (iv), RF power is applied to the reaction space in two occurrences between which a purging step is conducted (in some embodiments, three or more occurrences per cycle). In some embodiments, in step (iv), RF power applied to the reaction space is about 0.028 W/cm² to about 0.28 W/cm², preferably 0.07 W/cm² to about 0.21 W/cm². When a combination of H₂ and Ar is used as a reactant gas, etching can occur at a low RF power. In some embodiments, the duration of a pulse of RF power is about 0.2 seconds to about 5 seconds, preferably about 0.5 seconds to about 1 second. The longer the duration of a pulse of RF power, the greater the reduction of thickness becomes. If application of RF power is divided into multiple sessions, the reduction of thickness can significantly be lowered. By adjusting the duration of RF power application and the number of RF power applications per cycle, the step coverage at the sidewall and on the flat surface can be desirably adjusted.

In some embodiments, the temperature during the process cycle is about 50° C. to about 500° C., preferably about 100° C. to about 300° C.

In some embodiments, the dielectric film is a film of SiC, SiCN, SiN, SiOCN, or SiO. For example, a SiN film can be deposited when a precursor having a Si—N bond such as silylamine compounds or aminosilane compounds is used, whereas a SiO film can be deposited when a precursor having a Si—O bond such as alkoxide compounds is used, even when only hydrogen gas and argon gas are used as a reactant gas.

In some embodiments, a sidewall coverage of the deposited dielectric film is about 80% or higher, typically about 80% to about 130%, wherein the sidewall coverage is defined as a ratio of thickness of film on a sidewall of the trench to thickness of film on a blanket surface of the trench.

In some embodiments, a method for increasing a sidewall coverage of a dielectric film deposited according to any deposition method disclosed herein is provided, wherein in step (i), the precursor is fed in a pulse having a first duration, in step (ii), the reactant gas is supplied at a first flow rate, and in step (iv), RF power is applied in a pulse having a first duration, and the dielectric film has a first sidewall coverage, said sidewall coverage being defined as a ratio of thickness of film on a sidewall of the trench to thickness of film on a blanket surface of the substrate, said method comprising: (a) setting a second duration of the pulse of the precursor in step (i), a second flow rate of the reactant gas in step (ii), and a second duration of the pulse of RF power in step (iv), wherein at least one of the second flow rate of the reactant gas and the second duration of the pulse of RF power is higher than the first flow rate of the reactant gas and the first duration of the pulse of RF power, respectively, and/or the second duration of the pulse of the precursor is shorter than the first duration of the pulse of the precursor; and (b) repeating steps (i) to (iv) using the second flow rate of the reactant gas and the second duration of the pulse of RF power, thereby depositing a dielectric film having a second sidewall coverage which is higher than the first sidewall coverage.

In some embodiments, the film is deposited by PEALD under conditions shown in Table 1 below.

TABLE 1

(the numbers are approximate) Conditions for Deposition Cycle	
Substrate temperature	50 to 500° C. (preferably 100 to 400° C.)
Pressure	100 to 1000 Pa (preferably 200 to 500 Pa)
Precursor pulse	0.1 to 3 Sec (preferably 0.1 to 1 Sec)
Precursor purge	0.3 to 10 Sec
Flow rate of reactant (continuous)	50 to 1000 sccm (preferably 100 to 300 sccm)
Carrier gas	e.g., argon
Dilution gas	e.g., argon
Flow rate of carrier/dilution gas (continuous)	1000 to 5000 sccm (preferably 2000 to 4000 sccm)
RF power (13.56 MHz) for a 300-mm wafer	50 to 200 W (preferably 50 to 150 W)
RF power pulse (total)	0.2 to 5 sec (preferably 0.5 to 1 sec)
Purge	0.1 to 2 Sec
Growth rate per cycle	0.005 to 0.08 nm/cycle
Total thickness [nm]	3 to 20 Nm

FIG. 2 illustrates a PEALD process sequence according to an embodiment of the present invention. In this disclosure, the width of each column does not necessarily represent the actual time length, and a raised level of the line in each row represents an ON-state whereas a bottom level of the line in each row represents an OFF-state. The deposition cycle includes steps of feeding a precursor to a reaction zone, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone in this order, wherein a purge/carrier gas is supplied continuously to the reaction zone throughout the entire steps of the deposition cycle, and a reactant gas for deposition is supplied continuously to the reaction zone throughout the entire steps of the deposition cycle. In the deposition cycle, steps of feeding the precursor, purging the reaction zone, applying RF power to the reaction zone, and purging the reaction zone can be repeated p times (p is an integer of 5 to 100, typically 8 to 50), depending on the target compositions and quality of the film, although repeating is not required.

In this disclosure, the word “continuously” refers to at least one of the following: without breaking a vacuum, without being exposed to air, without opening a chamber, as an in-situ process, without interruption as a step in sequence, and without changing main process conditions, depending on the embodiment. In some embodiments, an auxiliary step such as a delay between steps or other step immaterial or insubstantial in the context does not count as a step, and thus, the word “continuously” does not exclude an intervening auxiliary step.

FIG. 3 illustrates a PEALD process sequence according to another embodiment of the present invention. In this sequence, after the sequence illustrated in FIG. 2, the deposition cycle further includes steps of applying again RF power (“RF-2”) to the reaction zone, and purging the reaction zone in this order. In other words, application of RF power illustrated in FIG. 2 is divided into two sessions, i.e., the application of RF power comprises a first application of RF power, purging, and a second application of RF power. In some embodiments, the application of RF power may comprise more than two applications of RF power (e.g., three or four sessions). The first application and the subsequent application of RF power can be conducted in the same manner or in different manners, e.g., at different pulse durations (the first duration is shorter or longer than the second duration), different intensities (the first intensity is lower or higher than the second intensity), etc.

In the sequence illustrated in FIG. 2, the precursor is supplied in a pulse using a carrier gas which is continuously

supplied. This can be accomplished using a flow-pass system (FPS) wherein a carrier gas line is provided with a detour line having a precursor reservoir (bottle), and the main line and the detour line are switched, wherein when only a carrier gas is intended to be fed to a reaction chamber, the detour line is closed, whereas when both the carrier gas and a precursor gas are intended to be fed to the reaction chamber, the main line is closed and the carrier gas flows through the detour line and flows out from the bottle together with the precursor gas. In this way, the carrier gas can continuously flow into the reaction chamber, and can carry the precursor gas in pulses by switching the main line and the detour line. FIG. 1B illustrates a precursor supply system using a flow-pass system (FPS) according to an embodiment of the present invention (black valves indicate that the valves are closed). As shown in (a) in FIG. 1B, when feeding a precursor to a reaction chamber (not shown), first, a carrier gas such as Ar (or He) flows through a gas line with valves b and c, and then enters a bottle (reservoir) 20. The carrier gas flows out from the bottle 20 while carrying a precursor gas in an amount corresponding to a vapor pressure inside the bottle 20, and flows through a gas line with valves f and e, and is then fed to the reaction chamber together with the precursor. In the above, valves a and d are closed. When feeding only the carrier gas (noble gas) to the reaction chamber, as shown in (b) in FIG. 1B, the carrier gas flows through the gas line with the valve a while bypassing the bottle 20. In the above, valves b, c, e, and f are closed.

The precursor may be provided with the aid of a carrier gas. Since ALD is a self-limiting adsorption reaction process, the number of deposited precursor molecules is determined by the number of reactive surface sites and is independent of the precursor exposure after saturation, and a supply of the precursor is such that the reactive surface sites are saturated thereby per cycle. A plasma for deposition may be generated in situ, for example, in an ammonia gas that flows continuously throughout the deposition cycle. In other embodiments the plasma may be generated remotely and provided to the reaction chamber.

As mentioned above, each pulse or phase of each deposition cycle is preferably self-limiting. An excess of reactants is supplied in each phase to saturate the susceptible structure surfaces. Surface saturation ensures reactant occupation of all available reactive sites (subject, for example, to physical size or “steric hindrance” restraints) and thus ensures excellent step coverage. In some embodiments the pulse time of one or more of the reactants can be reduced such that complete saturation is not achieved and less than a monolayer is adsorbed on the substrate surface.

The process cycle can be performed using any suitable apparatus including an apparatus illustrated in FIG. 1A, for example. FIG. 1A is a schematic view of a PEALD apparatus, desirably in conjunction with controls programmed to conduct the sequences described herein, usable in some embodiments of the present invention. In this figure, by providing a pair of electrically conductive flat-plate electrodes 4, 2 in parallel and facing each other in the interior 11 (reaction zone) of a reaction chamber 3, applying HRF power (13.56 MHz or 27 MHz) 20 to one side, and electrically grounding the other side 12, a plasma is excited between the electrodes. A temperature regulator is provided in a lower stage 2 (the lower electrode), and a temperature of a placed thereon is kept constant at a given temperature. The upper electrode 4 serves as a shower plate as well, and reactant gas (and noble gas) and precursor gas are introduced into the reaction chamber 3 through a gas line 21 and a gas line 22, respectively, and through the shower plate 4.

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Additionally, in the reaction chamber 3, a circular duct 13 with an exhaust line 7 is provided, through which gas in the interior 11 of the reaction chamber 3 is exhausted. Additionally, a transfer chamber 5 disposed below the reaction chamber 3 is provided with a seal gas line 24 to introduce seal gas into the interior 11 of the reaction chamber 3 via the interior 16 (transfer zone) of the transfer chamber 5 wherein a separation plate 14 for separating the reaction zone and the transfer zone is provided (a gate valve through which a wafer is transferred into or from the transfer chamber 5 is omitted from this figure). The transfer chamber is also provided with an exhaust line 6. In some embodiments, the deposition of multi-element film and surface treatment are performed in the same reaction space, so that all the steps can continuously be conducted without exposing the substrate to air or other oxygen-containing atmosphere. In some embodiments, a remote plasma unit can be used for exciting a gas.

In some embodiments, in the apparatus depicted in FIG. 1A, the system of switching flow of an inactive gas and flow of a precursor gas illustrated in FIG. 1B (described earlier) can be used to introduce the precursor gas in pulses without substantially fluctuating pressure of the reaction chamber.

In some embodiments, a dual chamber reactor (two sections or compartments for processing wafers disposed closely to each other) can be used, wherein a reactant gas and a noble gas can be supplied through a shared line whereas a precursor gas is supplied through unshared lines.

A skilled artisan will appreciate that the apparatus includes one or more controller(s) (not shown) programmed or otherwise configured to cause the deposition and reactor cleaning processes described elsewhere herein to be conducted. The controller(s) are communicated with the various power sources, heating systems, pumps, robotics, and gas flow controllers or valves of the reactor, as will be appreciated by the skilled artisan.

The present invention is further explained with reference to working examples below. However, the examples are not intended to limit the present invention. In the examples where conditions and/or structures are not specified, the skilled artisan in the art can readily provide such conditions and/or structures, in view of the present disclosure, as a matter of routine experimentation. Also, the numbers applied in the specific examples can be modified by a range of at least $\pm 50\%$ in some embodiments, and the numbers are approximate.

EXAMPLES

A silicon carbide film was formed on a Si substrate (0300 mm) having trenches with an aspect ratio of 3.5 (a width of 30 nm, and a depth of 110 nm) by PEALD using a sequence illustrated in FIG. 2 or 3, one cycle of which was conducted under the common conditions shown in Table 2 (process cycle) below using the PEALD apparatus illustrated in FIG. 1A and a gas supply system (FPS) illustrated in FIG. 2 with the specific conditions and sequence indicated in Table 3.

TABLE 2

(the numbers are approximate) Common Conditions for Process Cycle	
Substrate temperature	300.° C.
Pressure	300 Pa
Carrier gas and dilution gas	Ar
Reactant gas	H ₂

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TABLE 2-continued

(the numbers are approximate) Common Conditions for Process Cycle	
Flow rate of carrier/dilution gas (continuous)	2000 sccm/1000 sccm (3000 sccm total)
Purge after precursor feed pulse	1 sec
Purge after RF power pulse	1 sec
RF power	100 W
Target Thickness (nm)	10.

TABLE 3

(the numbers are approximate)						
	Sequence	Feed (seconds)	Feed amount (g/cycle)	RF pulse (seconds)	Precursor	H ₂ flow (sccm)
*1	FIG. 2	1	0.04	1	DVDMS	0
*2	FIG. 2	0.1	0.004	1	DVDMS	800
*3	FIG. 2	1.0	0.002	1	HMDS	0
4	FIG. 2	1	0.002	1	HMDS	50
5	FIG. 2	0.8	0.0016	1	HMDS	50
6	FIG. 2	0.6	0.0012	1	HMDS	50
7	FIG. 2	0.5	0.001	1	HMDS	50
8	FIG. 2	0.1	0.0002	1	HMDS	50
9	FIG. 2	0.5	0.001	0.5	HMDS	50
10	FIG. 2	0.1	0.0002	0.5 each	HMDS	50
11	FIG. 2	0.1	0.004	0.5 each	DVDMS	50
12	FIG. 2	0.1	0.004	1	DVDMS	50
13	FIG. 2	0.1	0.004	1	DVDMS	300

In Table 3, the Example numbers with “*” indicate comparative examples. Each obtained film was evaluated. Table 4 shows the results of evaluation. DVDMS is divinyl dimethylsilane, and HMDS is hexamethyldisilane.

TABLE 4

(the numbers are approximate)			
	GPC (nm/cycle)	Sidewall Coverage @AR3 (%)	Remarks
*1	0.03	35%	No damage
*2	—	—	Great damage
*3	0.12	40%	No damage
4	0.07	80%	No damage
5	0.05	85%	No damage
6	0.02	94%	No damage
7	0.009	125%	No damage
8	0.006	130%	Slight damage
9	0.007	130%	No damage
10	0.004	150%	No damage
11	0.06	85%	No damage
12	0.02	115%	No damage
13	0.01	130%	No damage

In Table 4, “GPC” represents growth rate per cycle, “Sidewall Coverage@AR3” represents a percentage of thickness of film deposited on a sidewall relative to thickness of film deposited on a blanket surface at a trench having an aspect ratio of 3, and “Remarks” describes damage observed on a surface of an underlying layer after deposition.

In the above examples, when no hydrogen gas was used as a reactant in Example 1, the sidewall coverage was significantly low, since no hydrogen gas was used, and sufficient argon plasma did not reach the sidewall of the trench, whereby sufficient active sites on the silicon-containing hydrocarbon precursor were not formed at the sidewall, i.e., the sidewall coverage was poor (35%). On the

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other hand, when excessive hydrogen gas (800 sccm) was used in Example 2, etching took place particularly on the top surface, and no film was deposited. However, when an appropriate quantity of hydrogen gas was used in Examples 12 (50 sccm) and 13 (300 sccm), etching desirably took place on the top surface, thereby interfering with deposition of film on the top surface and significantly increasing the sidewall coverage (115% in Example 12; 130% in Example 13). Further, when RF power was applied in two sessions in Example 11, the etching effect by hydrogen gas became weaker, and deposition by hydrogen gas was promoted. As a result, the GPC was increased, especially at the sidewall, thereby increasing the sidewall coverage (85%) as well as the GPC (0.06 nm/cycle), as compared with Example 12 (the sidewall coverage was 115%, and the GPC was 0.02 nm/cycle) and Example 1 (the sidewall coverage was 35%, and the GPC was 0.03 nm/cycle). The above differences between Examples 11 and 12 are opposite to those between Examples 8 and 10 discussed below. This may be because in Examples 11 and 12, the precursor possessed a carbon double bond (vinyl), and when the double bond was opened by a hydrogen plasma, chemisorption and formation of dangling bonds could have been promoted, more than etching.

When the duration of feed pulse was increased from 0.1 second (Example 8) to 0.5 seconds (Example 7), 0.6 seconds (Example 6), 0.8 seconds (Example 5), and 1.0 seconds (Example 4), the GPC was increased accordingly from 0.006 nm/cycle (Example 8) to 0.009 nm/cycle (Example 7), 0.02 nm/cycle (Example 6), 0.05 nm/cycle (Example 5), and 0.07 nm/cycle (Example 4), whereas the sidewall coverage was decreased accordingly from 130% (Example 8) to 125% (Example 7), 94% (Example 6), 85% (Example 5), and 80% (Example 4). When comparing Example 4 (with 50 sccm of hydrogen gas) and Example 3 (with 0 sccm of hydrogen gas), the GPC was lower in Example 4 (0.07 nm/cycle) than that in Example 3 (0.12 nm/cycle), the sidewall coverage was significantly higher in Example 4 (80%) than that in Example 3 (40%) due to the etching effect of a hydrogen plasma which was more prominent on the blanket surface than the sidewall. When the duration of feed pulse was as short as 0.1 second in Example 8, fine damage on the surface of the substrate was observed. Further, when the duration of RF power pulse was shorter in Example 9 (0.5 seconds) than in Example 8 (1.0 second), the GPC was slightly increased whereas the sidewall coverage was substantially unchanged. Further, when RF power was applied in two sessions in Example 10 as compared with Example 8 (one session), the etching effect by hydrogen gas became stronger, and deposition by hydrogen gas was least promoted. As a result, the GPC was low (0.004 nm/cycle), as compared with Example 8 (0.006 nm/cycle). The above differences between Examples 8 and 10 are opposite to those between Examples 11 and 12 discussed above. This may be because in Examples 8 and 10, the precursor did not possess a carbon double bond, and the etching effect of a hydrogen plasma became more prominent by two-session application, resulting in lower GPC in Example 10 (0.004 nm/cycle) than that in Example 8 (0.006 nm/cycle), and higher sidewall coverage in Example 10 (150%) than that in Example 8 (130%).

Accordingly, it was confirmed that by adjusting the duration of feed pulse, the duration of RF power pulse, and/or the flow rate of hydrogen gas, the sidewall coverage and the GPC can be desirably adjusted.

It will be understood by those of skill in the art that numerous and various modifications can be made without departing from the spirit of the present invention. Therefore,

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it should be clearly understood that the forms of the present invention are illustrative only and are not intended to limit the scope of the present invention.

We claim:

1. A method for forming a dielectric film in a trench on a substrate by plasma-enhanced atomic layer deposition (PEALD) performing one or more process cycles, each process cycle comprising:

(i) feeding a silicon-containing precursor in a pulse to a reaction space where the substrate is placed, said silicon-containing precursor being constituted by one or more hydrocarbon-containing compounds selected from the group consisting of: SiH_3R_2 , $\text{Si}_2\text{H}_2\text{R}_4$, SiR_2X_2 , Si_2R_6 , SiH_3R , $\text{Si}_2\text{H}_4\text{R}_2$, SiH_2RX , $\text{C}_3\text{H}_6\text{SiH}_2$, $\text{C}_2\text{H}_4\text{SiH}_2$, $\text{C}_2\text{H}_4\text{Si}_2\text{H}_2$, $\text{SiNHHSiR}_4\text{H}_2$, SiNHHSiR_6 , and SiHX_2R , wherein each X is independently chain or cyclic C_xH_y , and each R is independently chain or cyclic C_xH_y , cyclic $\text{N}_x\text{C}_y\text{H}_z$, $\text{N}(\text{C}_x\text{H}_y)_2$, $\text{N}(\text{C}_x\text{H}_y)\text{H}$, $\text{O}(\text{C}_x\text{H}_y)$, or OH, wherein x, y, and z are integers;

(ii) supplying a hydrogen-containing reactant gas to the reaction space at a flow rate of more than about 30 sccm but less than about 800 sccm in the absence of nitrogen-containing gas;

(iii) supplying a noble gas to the reaction space; and

(iv) applying RF power to the reaction space in the presence of the hydrogen-containing reactant gas and the noble gas and in the absence of any precursor in the reaction space, to form a monolayer constituting a dielectric film on a substrate at a growth rate of less than one atomic layer thickness per cycle.

2. The method according to claim 1, wherein the growth rate of the monolayer is less than 0.1 nm/cycle.

3. The method according to claim 1, wherein the precursor has a chemical formula where at least one of X or R is an unsaturated hydrocarbon.

4. The method according to claim 1, wherein the precursor has a cyclic structure.

5. The method according to claim 1, wherein the silicon-containing precursor is one or more compounds selected from the group consisting of: dimethylsilane, divinylsilane, dipyrityldisilane, tertamethyldisilane, divinyltrimethylsilane, dimethyldipyrityldisilane, hexamethyldisilane, silylacetylene, allylsilane, divinyltrisilane, dimethyldisilane, vinylmethylsilane, silacyclobutane, silacycloethane, di silacycloethane, tetramethyldisilazane, hexamethyldisilazane, and dimethyldiprilyldisilane.

6. The method according to claim 1, wherein the reactant gas is hydrogen gas.

7. The method according to claim 1, wherein the noble gas is argon.

8. The method according to claim 1, wherein the reactant gas is supplied continuously to the reaction space throughout each process cycle.

9. The method according to claim 1, wherein the noble gas is continuously supplied to the reaction space throughout the process cycle.

10. The method according to claim 1, wherein a ratio of flow rate of the noble gas to flow rate of the reactant is about 10:1 to about 60:1.

11. The method according to claim 1, wherein in step (iii), the noble gas is supplied to the reaction space at a flow rate of about 1,000 sccm to about 5,000 sccm.

12. The method according to claim 1, wherein in step (iv), no gas other than the reactant gas and the noble gas is supplied to the reaction space.

13. The method according to claim 1, wherein in step (i), the silicon-containing precursor is fed in an amount of about

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0.00002 g/cycle to about 0.01 g/cycle and in a pulse having a duration of about 0.1 seconds to about 1.0 second.

14. The method according to claim 1, wherein each process cycle further comprises a purging step between steps (i) and (iv), and between steps (iv) and (i) when the process cycle is repeated.

15. The method according to claim 1, wherein in step (iv), RF power is applied to the reaction space in two occurrences between which a purging step is conducted.

16. The method according to claim 1, wherein in step (iv), RF power applied to the reaction space is about 0.028 W/cm² to about 0.28 W/cm².

17. The method according to claim 1, wherein the temperature during the process cycle is about 50° C. to about 500° C.

18. The method according to claim 1, wherein the dielectric film is a film of SiC, SiCN, SiN, SiOCN, or SiO.

19. The method according to claim 1, wherein a sidewall coverage of the deposited dielectric film is about 80% or higher, wherein the sidewall coverage is defined as a ratio of thickness of film on a sidewall of the trench to thickness of film on a blanket surface of the trench.

20. A method for increasing a sidewall coverage of a dielectric film deposited according to claim 1 wherein in

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step (i), the precursor is fed in a pulse having a first duration, in step (ii), the reactant gas is supplied at a first flow rate, and in step (iv), RF power is applied in a pulse having a first duration, and the dielectric film has a first sidewall coverage, said sidewall coverage being defined as a ratio of thickness of film on a sidewall of the trench to thickness of film on a blanket surface of the trench, said method comprising:

(a) setting a second duration of the pulse of the precursor in step (i), a second flow rate of the reactant gas in step (ii), and a second duration of the pulse of RF power in step (iv), wherein at least one of the second flow rate of the reactant gas and the second duration of the pulse of RF power is higher than the first flow rate of the reactant gas and the first duration of the pulse of RF power, respectively, and/or the second duration of the pulse of the precursor is shorter than the first duration of the pulse of the precursor; and

(b) repeating steps (i) to (iv) using the second flow rate of the reactant gas and the second duration of the pulse of RF power, thereby depositing a dielectric film having a second sidewall coverage which is higher than the first sidewall coverage.

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